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I also certify that the attached copy of the request for grant of a Patent (Form 1/77) bears a correction, effected by this office, following a request by the applicant and agreed to by the Comptroller-General.

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Dated 9 January 2004

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GB 0300374.6

By virtue of a direction given under Section 30 of the Patents Act 1977, the application is proceeding in the name of:

SÜD-CHEMIE AG,
Lenbachplatz 6,
D-80333 MÜNCHEN,
Federal Republic of Germany

Incorporated in the Federal Republic of Germany,

[ADP No. 06478044001]

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JAK/6468-GB

2. Patent application number
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0300374.6

08 JAN 2003

3. Full name, address and postcode of the or of each applicant *(underline all surnames)*

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Patents ADP number *(if you know it)*

8537265001

If the applicant is a corporate body, give the country/state of its incorporation

Canada

SECTION 30 (1977 ACT) APPLICATION FILED 25/11/03

RE (F) 6.2.03

4. Title of the invention

Organoclay Compositions

5. Name of your agent *(if you have one)*

"Address for service" in the United Kingdom to which all correspondence should be sent *(including the postcode)*

Stevens Hewlett & Perkins
1 St. Augustine's Place
Bristol BS1 4UD
UK

Patents ADP number *(if you know it)*

1545002

6. If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or of each of these earlier applications and *(if you know it)* the or each application number

Country	Priority application number *(if you know it)*	Date of filing *(day / month / year)*

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Number of earlier application	Date of filing *(day / month / year)*

8. Is a statement of inventorship and of right to grant of a patent required in support of this request? *(Answer 'Yes' if:*
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b) there is an inventor who is not named as an applicant, or
c) any named applicant is a corporate body.
See note (d))

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Patents Form 1/77

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Claim (s)	6 8
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Request for preliminary examination and search *(Patents Form 9/77)*	-
Request for substantive examination *(Patents Form 10/77)*	-
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11. I/We request the grant of a patent on the basis of this application.

Signature Stevens Hewlett and Perkins Date 8 January 2003

Stevens Hewlett & Perkins

12. Name and daytime telephone number of person to contact in the United Kingdom

J A Knowles 0117 9226007

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ORGANOCLAY COMPOSITIONS

The present invention relates to organoclay compositions, to polymer compositions comprising organoclays, to processes for the production of polymer compositions and to uses of organoclay compositions as flame retardants, smoke suppressants or char promoters in polymer compositions.

Inorganic layered materials, for example smectite clays, may be intercalated with ions or other charged or uncharged species. Large species intercalated in layered materials may result in a pillared structure or, under the right conditions, exfoliation of the layers. Completely exfoliated smectite clays, for example montmorillonite, may have particle sizes with high aspect ratios of about 100 nm to 500 nm with a layer thickness of about 1 nm.

Layered materials, especially clays, intercalated with long chain ions or other charged species are known as organoclays. With further processing, organoclays may be exfoliated, resulting in particles having at least one dimension on the nanometer scale, for this reason, organoclays are sometimes referred to as nanoclays. Organoclays, for example those supplied under the trade name Nanofil by Süd-Chemie AG, are available commercially (equivalent types are also available from Southern Clay Products, Nanocor, Elementis and Ausimont). Nanofil is produced from natural or synthetic high purity layered silicates based on montmorillonite, intercalated (in the case of Nanofil 15) with distearyldimethyl ammonium chloride. Nanofil 15, as supplied, has a median particle size, d50, of about 30 to 80μm and an upper particle size limit of 300μm. EA 108 from Elementis is based on hectorite. EA 108 shows a much higher whiteness than its competitors and a much reduced layer thickness as specified in the technical literature available from Elementis Corp. USA.

Organoclays may be used in fillers for polymer products. WO 00/66657 describes a polymer composition comprising a polymer and a synergistic flame retardant additive combination which comprises a nanoclay and a

second filler, preferably aluminium trihydroxide (ATH) or magnesium hydroxide.

Organoclays are useful additives, particularly in zero halogen flame retardants (often known as OHFR), for example, ATH, or magnesium hydroxide because they promote the formation of a stable char layer during combustion of a polymer. The formation of a char layer creates a barrier to further ignition and also reduces slumping or dripping of flaming polymer, which can promote the progression of a flame.

Organoclays as marketed to date, in combination with OHFR fillers, do, however, worsen the mechanical and extrusion properties of polymer compositions. They create problems during incorporation into the polymer formulation, besides being extremely difficult to exfoliate and therefore to distribute evenly throughout the polymer matrix. Additional problems are electrical, especially in salt-water immersion tests (for example, with 300 V DC being applied for several days until arcing occurs as the final failure). A solution to overcome these problems has been to use surface modified OHFR fillers.

The present invention aims to mitigate these problems by providing pre-treated organoclays for use in compositions.

The present invention accordingly provides, in a first aspect, an organoclay composition comprising an exfoliation-promoted organoclay.

The organoclay will usually have a median particle size (at least in one dimension) in the range 0.1 to 1000 μm, preferably 0.1 to 100 μm and more preferably 1 to 15 μm, and most preferably 2 to 10μm

The inventors have surprisingly found that exfoliation of the organoclay can be enhanced (i.e. promoted) by using an appropriate additive or by milling the organoclay or more preferably a combination of both.

The exfoliation-promoted organoclay may comprise milled organoclay. This is advantageous because milling is a well-known process enabling high throughput and, therefore, high yields of precursor material. The milling process may use a mill selected from jet mills, ball mills, vibrating mills, roll mills, hammer mills, impact mills, pin mills or attrition mills. Each milling

method has its own particular advantages and disadvantages which will be known to the skilled person.

Milled organoclay may be added to a polymer mix by placing the milled organoclay in a container of suitable composition, for example a container of the same composition as the polymer to which the organoclay is to be added or of a polymer compatible with the polymer to which the organoclay is to be added. Use of such a container mitigates the dust that may arise when using milled organoclays. The preferred form of container is a sealable bag. Alternatively, milled organoclay may be incorporated in a masterbatch together with fillers for use in polymer compositions.

A masterbatch is commonly known in the industry to mean a polymeric composition having a high percentage of additive or additives, (as well as possibly fillers, pigments etc.) to facilitate the manufacture of polymers with specific properties. Masterbatches are usually added in small quantities to large volumes of the same base polymer or one compatible with the base polymer to achieve desired properties.

Surprisingly, milled organoclay, particularly when incorporated in a masterbatch together with zero halogen fillers for use in polymer compositions, enhances the mechanical and processing properties of the polymer with an improved or with little negative effect on the beneficial flame-retardant properties of such compositions. This is advantageous because the improved mechanical and processing properties enable a higher loading of flame retardant to be used for substantially the same mechanical/processing properties. Alternatively, a lower loading of flame retardant may be used with consequential improved mechanical/processing properties and yet with little or no effect on the flame retardant properties of the polymer composition.

The inventors have also found that the exfoliation of organoclays can be enhanced by the use of suitable additives. Thus, preferably, the exfoliation-promoted organoclay comprises an organoclay and an exfoliation-promoting additive.

In a preferred embodiment, the exfoliation-promoted organoclay comprises a milled organoclay and at least one exfoliation-promoting additive in combination.

An advantage of using an exfoliation-promoting additive either on its own or in combination with the milled organoclay is that it mitigates problems that may arise with milled organoclay on its own. In particular, milled organoclay, being a finely divided powder, generates dust and is a potential explosion hazard.

The applicants have found that mixing the milled organoclay powder with an additive before introducing the mixture to a polymer is a preferred method of manufacture, and mitigates this problem.

The exfoliation-promoting additive may comprise:

(a) at least one fatty acid derivative selected from polymeric fatty acids, keto fatty acids, fatty alkyl oxazolines and fatty alkylbisoxazolines, or

(b) at least one fatty acid derivative selected from polymeric fatty acids, keto fatty acids, fatty alkyl oxazolines and fatty alkylbisoxazolines, at least one siloxane derivative selected from oligoalkylsiloxanes, polydialkylsiloxanes, polyalkylarylsiloxanes, polydiarylsiloxanes, oligoalkylsiloxanes which have been functionalized with at least one reactive group, polydialkylsiloxanes which have been functionalized with at least one reactive group, polyalkylarylsiloxanes which have been functionalized with at least one reactive group and polydiarylsiloxanes which have been functionalized with at least one reactive group, or

(c) at least one fatty acid, at least one siloxane derivative selected from oligoalkylsiloxanes, polydialkylsiloxanes, polyalkylarylsiloxanes, polydiarylsiloxanes, oligoalkylsiloxanes which have been functionalized with at least one reactive group, polydialkylsiloxanes which have been functionalized with at least one reactive group, polyalkylarylsiloxanes which have been functionalized with at least one reactive group, and polydiarylsiloxanes which have been functionalized with at least one reactive group.

The exfoliation-promoting additive may, alternatively, comprise at least one fatty acid, or a salt, hydrogenated derivative, alcohol derivative, amine derivative or other derivative thereof. This is advantageous because the chemical compatibility with the intercalating agents commonly used is improved and at the same time the cost is reduced.

The fatty acid usually comprises a fatty acid having from 10 to 30 carbon atoms, and preferably comprises a mono- or polyunsaturated hydroxy fatty acid.

In a preferred embodiment, the exfoliation-promoting additive comprises a fatty acid selected from lauric, palmitic, stearic, oleic, lineolic and capric acid, or castor bean oil. These additives are advantageous because they are readily available in bulk quantities, relatively safe to use on an industrial scale and cost-effective.

The exfoliation-promoting additive may alternatively comprise a fatty acid derivative selected from polymeric fatty acids, keto fatty acids, fatty alkyl oxazolines and fatty alkyl bisoxazolines.

As an alternative, the exfoliation-promoting additive may be selected from a liquid ethylene-propylene copolymer (EPM), a liquid ethylene-propylene terpolymer (EPDM) and a mixture thereof, the liquid EPM copolymers having a mean molecular weight of less than 20,000, and the liquid EPDM terpolymer having a mean molecular weight of less than 20,000, the ratio of ethylene to propylene in said ethylene-propylene copolymer and in said ethylene-propylene terpolymer being 40:60 to 60:40, a thermoplastic elastomer, a coupling agent, a crosslinking agent and mixtures thereof.

An important use of the organoclay compositions of the invention is as, or in, fillers in polymer compositions. Thus, the present invention provides in a second aspect a polymer composition comprising an organoclay composition according to the first aspect and a polymer (or a combination of suitable polymers). Suitable polymers include polymers selected from polyethylene-vinyl acetate co-polymer (EVA), ethylene ethylacrylate copolymer (EEA), ethylene methylacrylate co-polymer (EMA), ethylene butylacrylate co-polymer (EBA) and the maleic anhydride (MAH) grafted derivatives thereof, and

ionomers (e.g. AC types from Allied Colloids), styrene elastomer systems (i.e. compositions) as specified by SBS Kraton D from Shell, SIS and SEBS (Kraton G from Shell), ether ester block copolymers (EEBC) as specified by HYTREL from DuPont, polyether polyamide block copolymers (PEBA) as specified in PEBAX from Atofina, TPE blends e.g. EVA/SAN ; EVA/ PA 11; EVA/PS, PVDF/EVA as specified by ALCRYN from DuPont, TPUs (thermoplastic polyurethane elastomers) from Bayer, and TPQs (thermoplastic silicone rubber) from Wacker.

For use in flame retardant compounds, especially in zero halogen flame retardant (OHFR) compounds, the organoclay composition will usually be used in combination with appropriate fillers or mixtures of fillers. Thus, preferably, the polymer composition further comprises an inorganic filler material. Examples of inorganic filler materials are materials comprising aluminium oxyhydroxide, aluminium hydroxide, magnesium hydroxide, magnesium oxide, brucite, magnesium carbonate, hydromagnesite, huntite, boehmite, bauxite, calcium carbonate, talc, glass powder, hydrotalcite, Dawsonite, borate, especially zinc borate, stannate, and hydroxystannate, especially the zinc salt, phosphate, e.g. melamine-phosphate or ammonium phosphate. An advantage of using organoclay compositions in combination with a filler is that the need to use surface modified filler (e.g. ATH or magnesium hydroxide) to achieve an acceptable level of mechanical or electrical properties is reduced.

The most preferred inorganic fillers comprise aluminium hydroxide (alumina trihydrate ATH, having a formula $Al(OH)_3$), and magnesium hydroxide in its various forms.

In addition to being an effective flame retardant, aluminium hydroxide is also widely used for its smoke suppressant characteristics.

Magnesium hydroxide, $Mg(OH)_2$, for example, the naturally occurring form brucite is also very effective as a flame retardant and smoke suppressant and can be used where the polymer is processed at temperatures above 180°C.

Magnesium hydroxide (e.g. brucite) may also be used in applications where the temperature of the finished article is above 150°C for a prolonged period of time.

The polymer composition may alternatively comprise a halogenated polymer, for example polyvinyl chloride (PVC), and a compound using a halogenated (in particular brominated) flame retardant or a flame retardant composition, frequently in combination with antimony trioxide.

A particular advantage of using the inventive organoclay composition in a polymer composition is that the organoclay composition acts as a compatibilizer and char promoter and thus as an effective flame retardant.

The polymer composition may also comprise a stabiliser.

The present invention provides in a further aspect a process for the production of a polymer composition comprising

a) providing an organoclay
b) treating the organoclay to promote exfoliation
c) mixing the exfoliation-promoted organoclay with a polymer
d) optionally introducing an OHFR filler

The step b) of treating the organoclay to promote exfoliation may comprise milling the organoclay or it may comprise mixing the organoclay with an exfoliation promoted additive, or it may comprise a combination of both.

The step c) of mixing the exfoliation-promoted organoclay with a polymer may comprise mixing the exfoliation-promoted organoclay with a polymer to form a masterbatch which can then be introduced downstream to the same or another polymer or it may comprise mixing the exfoliation-promoted organoclay composition directly with the final polymer compound.

The sequence of process steps may vary. For example the optional introduction of the OHFR filler in step d) may take place towards the end of the process or it may occur at an earlier stage. An example would be where the organoclay powder and additive combination is mixed with the OHFR filler and subsequently compounded with the polymer system into the final OHFR compound or the organoclay and additive combination and OHFR filler(s) are

dosed separately into a stream of molten compound polymer to produce the final OHFR compound.

A preferred process route comprises the following steps:

a) provide an organoclay
b) mill the organoclay to promote exfoliation
c) mix the milled organoclay from b) with an exfoliation-promoting additive or additives
d) combine the mixture from c) with a polymer to form a masterbatch
e) mix the masterbatch from d) with the same or a compatible polymer in a compounding machine
f) compound the mixture from e)
g) use the compounded mixture from f) in a further suitable processing machine and when the polymer composition from f) is molten add OHFR filler material to create the final OHFR product.

As mentioned above the sequence of processing steps may vary with regard to the stage in the process at which OHFR fillers are introduced. An alternative process to the one described above would be to introduce the OHFR filler at the same time that the milled organoclay is mixed with exfoliation-promoting additive or additives at step c). Then this mixture of milled organoclay, exfoliation-promoting additive and OHFR filler would be combined with a polymer to form the masterbatch.

Such a process is advantageous because treating the organoclay to promote exfoliation before the compounding step improves the overall efficiency of the compounding and less energy and less time are required to exfoliate the organoclay in the polymer matrices. This is of particular benefit because it reduces the residence time of the polymer, keeping the thermal history (i.e. the time over which it is at an elevated temperature) of the polymer to a minimum and counteracting thermal degradation which adversely effects colour and mechanical performance of the compound. The throughput is increased and this increases the cost effectiveness of the compounding step.

The process of the invention is also advantageous because it enables a reduction in the overall load of organoclay in the polymer composition, preferably to below a level of two parts and more preferably to a level below one part in a hundred, without compromising the flame retardant properties. This compares to the conventional loading of between five and ten parts organoclay per hundred parts of compound, (parts are weight based).

It is thought that treating the organoclay to promote exfoliation before compounding helps to intimately mix the organoclay with e.g. OHFR- filler and polymer during the compounding step. This has a significant effect on e.g. the mechanical properties of the compounded polymer composition. For example, polymer systems are obtainable with an elongation at break of more than 200% (equivalent to 2 m/m) and preferably of 500% (equivalent to 5 m/m) or more compared to 10 % (equivalent to 0.1 m/m) without the use of this invention.

The process may further comprise extruding the polymer composition (e.g. for use in cabling or ducting) or injection moulding the polymer composition, blow-film extrusion or rotary moulding into the final article.

Organoclay compositions according to the invention are advantageously used as flame retardant synergists, smoke suppressants, char promoters and compatibilizers as well as performance enhancing agents and barrier forming agents in polymer compositions. Thus, the invention further provides a polymer article comprising an organoclay composition according to the invention, the article being in the form of a cable, trunking, ducting or a hose.

CLAIMS

1. An organoclay composition comprising an exfoliation-promoted organoclay.

2. An organoclay composition as claimed in claim 1, wherein the median particle size of the organoclay is in the range 0.1 to 1000 μm.

3. An organoclay composition as claimed in claim 2, wherein the median particle size of the organoclay is in the range 1 to 15μm.

4. An organoclay composition as claimed in claim 3, wherein the median particle size of the organoclay is in the range 2 to 10μm.

5. An organoclay composition as claimed in any of the preceding claims wherein the exfoliation-promoted organoclay comprises milled organoclay.

6. An organoclay composition as claimed in any of the preceding claims, wherein the exfoliation-promoted organoclay comprises an organoclay and an exfoliation-promoting additive.

7. An organoclay composition as claimed in any of the preceding claims wherein the exfoliation-promoted organoclay comprises a milled organoclay mixed with an exfoliation-promoting additive.

8. An organoclay composition as claimed in either claim 6 or claim 7, wherein the exfoliation promoting additive comprises:
   (a) at least one fatty acid derivative selected from polymeric fatty acids, keto fatty acids, fatty alkyl oxazolines and fatty alkylbisoxazolines, or

(b) at least one fatty acid derivative selected from polymeric fatty acids, keto fatty acids, fatty alkyl oxazolines and fatty alkylbisoxazolines, at least one siloxane derivative selected from the group consisting of oligoalkylsiloxanes, polydialkylsiloxanes, polyalkylarylsiloxanes, polydiarylsiloxanes, oligoalkylsiloxanes which have been functionalized with at least one reactive group, polydialkylsiloxanes which have been functionalized with at least one reactive group, polyalkylarylsiloxanes which have been functionalized with at least one reactive group and polydiarylsiloxanes which have been functionalized with at least one reactive group,

or

(c) at least one fatty acid, at least one siloxane derivative selected from oligoalkylsiloxanes, polydialkylsiloxanes, polyalkylarylsiloxanes, polydiarylsiloxanes, oligoalkylsiloxanes which have been functionalized with at least one reactive group, polydialkylsiloxanes which have been functionalized with at least one reactive group, polyalkylarylsiloxanes which have been functionalized with at least one reactive group, and polydiarylsiloxanes which have been functionalized with at least one reactive group.

9. An organoclay composition as claimed in either claim 6 or claim 7, wherein the exfoliation-promoting additive comprises a fatty acid, or a salt, hydrogenated derivative, alcohol derivative, amine derivative or other derivative thereof.

10. An organoclay composition as claimed in claim 9, wherein the fatty acid has 10 to 30 carbon atoms.

11. An organoclay composition as claimed in claim 9, wherein the fatty acid comprises a mono- or polyunsaturated hydroxy fatty acid.

12. An organoclay composition as claimed in either claim 6 or claim 7, wherein the exfoliation promoting additive is a fatty acid selected from lauric, palmitic, stearic, oleic, lineolic, and capric acids, or a castor bean oil.

13. An organoclay composition as claimed in either claim 6 or claim 7, wherein the exfoliation-promoting additive comprises a fatty acid derivative selected from polymeric fatty acids, keto fatty acids, fatty alkyl oxazolines and fatty alkyl bisoxazolines.

14. An organoclay composition as claimed in either claim 6 or claim 7, wherein the exfoliation promoting additive is selected from a liquid ethylene-propylene copolymer (EPM), a liquid ethylene-propylene terpolymer (EPDM) and a mixture thereof, the liquid EPM copolymers having a mean molecular weight of less than 20,000, and the liquid EPDM terpolymer having a mean molecular weight of less than 20,000, the ratio of ethylene to propylene in said ethylene-propylene copolymer and in said ethylene-propylene terpolymer being 40:60 to 60:40, a coupling agent, a crosslinking agent and mixtures thereof.

15. A polymer composition comprising an organoclay composition as claimed in any one of the preceding claims and a polymer.

16. A polymer composition as claimed in claim 15, wherein the polymer is selected from polyethylene-vinyl acetate co-polymer, ethylene ethylacrylate copolymer, ethylene methylacrylate co-polymer, ethylene butylacrylate co-polymer and the maleic anhydride grafted derivatives thereof, ionomers, styrene elastomer systems, SIS, SEBS, ether ester block copolymers, polyamide block copolymers, TPE blends, thermoplastic polyurethane elastomers and thermoplastic silicone rubber.

17. A polymer composition comprising an organoclay composition as claimed in any of claims 1 to 14, a matrix polymer system and an inorganic filler material.

18. A polymer composition as claimed in either of claims 15 or 16, further comprising an inorganic filler material.

19. A polymer composition as claimed in claim 17, wherein the inorganic filler material comprises aluminium oxyhydroxide, aluminium hydroxide, magnesium hydroxide, magnesium oxide, brucite, magnesium carbonate, hydromagnesite, huntite, boehmite, bauxite, calcium carbonate, talc, glass powder, a hydrotalcite, Dawsonite, a borate, especially zinc borate, a stannate, a hydroxystannate, a zinc salt, or a phosphate, especially melamine-phosphate or ammonium phosphate

20. A polymer composition as claimed in claim 17 wherein the inorganic filler is aluminium hydroxide

21. A polymer composition as claimed in claim 17 wherein the inorganic filler is magnesium hydroxide.

22. A polymer composition as claimed in claim 21 wherein the inorganic filler comprises surface-treated magnesium hydroxide.

23. A polymer composition as claimed in any of claims 18 to 22 further comprising a stabilizer and/or a colourant.

24. A polymer composition comprising polyvinylchloride polymer containing an organoclay composition as claimed in any of claims 1 to 14 and a suitable flame retardant system.

25. A polymer composition as claimed in claim 24 wherein the flame retardant system comprises antimony trioxide.

26. A polymer composition comprising a polymer selected from polyolefins, polyesters, polyamides, thermoplastic elastomers and their copolymers and alloys that are flame-retarded by means of a halogenated compound and an organoclay composition as claimed in any of claims 1 to 14.

27. A process for the production of a polymer composition, the process comprising,
providing an organoclay,
treating the organoclay to promote exfoliation, and
mixing the exfoliation-promoted organoclay with a polymer or mixture of polymers.

28. A process as claimed in claim 27 wherein treating the organoclay to promote exfoliation comprises milling the organoclay.

29. A process as claimed in claim 27 wherein treating the organoclay to promote exfoliation comprises mixing the organoclay with an exfoliation-promoting additive.

30. A process as claimed in claim 27 wherein treating the organoclay to promote exfoliation comprises both milling the organoclay and mixing the organoclay with an exfoliation-promoting additive.

31. A process as claimed in any of claims 27 to 30, further comprising adding an OHFR filler.

32. A process as claimed in any of claims 27 to 31 further comprising a process step selected from compounding, extruding, injection moulding, blow film extruding or roto-moulding the polymer composition.

33. Use of a polymer composition incorporating an organoclay composition as claimed in any one of claims 1 to 14 as a flame retardant synergist, char promoter, barrier-forming or performance enhancing agent in a polymer composition, preferably in a polymer composition comprising a zero halogen flame-retardant compound containing aluminium hydroxide or magnesium hydroxide or mixtures of these.

34. A polymer article comprising an organoclay composition as claimed in any of claims 1 to 14, the article being in the form of a cable, trunking, ducting or a hose.

PCT Application
PCT/EP2004/000071